

October 22, 1999

William Eckroade
Department of Energy
Germantown, MD

The laboratory used the term "Yellow Cake" to describe a receiver ash sample taken from Building 410 (see sampled container on page 18 of the Phase I report).

W. A. Eckroade
11/5/99

Dear Mr. Eckroade:

The attached reports have plutonium, neptunium, and gamma results for the solid uranium compounds (the green solid and yellowcake) we received from Paducah.

In the green solid, uranium is a minor component – about 0.5%, according to the gamma count. The green color is caused largely by iron and other metals, not UF_4 . We could not detect plutonium or neptunium in it. The only gamma emitter detectable in the green solid was U-238.

The yellowcake is about 65% uranium. We measured about 3 pCi Pu-239 per gram, but that result is probably low. Our Pu-239 spikes on this material gave only about a 50% recovery, and the measured sample Pu-239 concentration is probably comparably low. (If necessary, we could measure the Pu-239 accurately with a different method, ignoring the Np-237.)

We detected very slight Np-237 in the yellowcake, but the Np-237 results may be biased high by U-234.

These unusual samples required a special analytical test plan. We digested them in a sulfuric-nitric-hydrochloric acid mixture in Teflon beakers to expel most of the fluoride and get the samples completely in solution. We dissolved large aliquots (35 grams of each green solid sample and 15 grams of each yellowcake sample), then diluted them to large volumes and withdrew small aliquots for analysis, in order to avoid sample homogeneity problems. The samples were not uniform in color and were probably not very homogeneous. The yellowcake had pieces of green solid, and both yellowcake and green solids had what looked like rotten wood. The samples eventually dissolved completely, rotten wood and all.

We counted aliquots of the solutions for gamma emitters. (Had we counted the solids directly for gamma emitters, their high density would have caused absorption of low energy gammas and inaccurate results.)

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For plutonium-neptunium analysis, we withdrew two aliquots of each sample solution. We spiked one of each duplicate pair with Pu-239 and Np-237, and left the other unspiked. (We expected to find Np-237, so we didn't use Pu-242 tracer, which has an alpha energy close enough to Np-237 to interfere at low Np-237 concentrations.) We evaporated the sample aliquots to white fumes with nitric and sulfuric acids to expel the fluoride, then used anion exchange in hydrochloric acid solution to isolate Np and Pu. We mounted the separated Np-Pu product for counting (procedure PNL-ALO-496), then measured the alpha emitters by alpha spectroscopy (procedure PNL-ALO-422).

Sincerely,

Chuck Soderquist

Battelle, Pacific Northwest National Laboratory
Richland, WA
Radiochemical Processing Group

filename 00-30
 10/21/99

Client: DOE-EH Cognizant Scientist: _____

Concur: _____

Procedure: Test plan, followed by alpha spectroscopy (procedure PNL-ALO-422).

Sample	Lab ID	Measured Activities, pCi per gram \pm counting error			pCi/g	
		Np-237 \pm 1s	Pu-239+240 \pm 1s	Pu-238 \pm 1s	Np-237 MDC	Pu-239 MDC
*DHW34008-99	00-30	0.00 \pm 0.00	0.07 \pm 0.04	0.11 \pm 0.06	0.1	0.2
*DHW340D08-99	00-30	0.10 \pm 0.04	0.04 \pm 0.03	0.17 \pm 0.08	0.1	0.2
**DHW41008-99	00-32	1.0 \pm 0.2	2.2 \pm 0.3	0.10 \pm 0.10	0.2	0.3
**DHW410D08-99	00-33	0.8 \pm 0.2	3.9 \pm 0.4	0.10 \pm 0.09	0.2	0.2
	Blank 1	0.02 \pm 0.02	0.00 \pm 0.01	0.00 \pm 0.01	0.1	0.1
	Blank 2	0.00 \pm 0.01	0.00 \pm 0.01	0.02 \pm 0.02	0.1	0.1

*green solid

**yellowcake

Results in shaded cells are below the detection limit

Yellowcake Pu-239 results are probably biased low (see spike results below)

	Observed			Expected	Recovery		Observed	Expected	Recovery
	dpm	Np-237	Np-237				dpm	Pu-239	Pu-239
Greensalt Spike 1	101.6	126	81%			43.2	50.1	86%	
Greensalt Spike 2	99.6	126	79%			44.0	50.1	88%	
Yellowcake Spike 1	109.0	126	87%			23.6	50.1	47%	
Yellowcake Spike 2	104.6	126	83%			29.4	50.1	59%	

Battelle, Pacific Northwest National Laboratory
 Richland, WA
 Radiochemical Processing Group

filename 00-30
 10/22/99

Client: DOE-EH Cognizant Scientist: _____

Concur: _____

Procedure: Gamma counting, procedure PNL-ALO-450

Sample	Lab ID	--- Gamma Emitters, pCi per liter \pm 1s ---				
		Co-60	Cs-137	U-235	U-238	Am-241
DHWEB34008-99	00-28	< 2.E+1	< 2.E+1	< 4.E+1	< 2.E+3	< 3.E+2
DHWEB41008-99	00-29	< 3.E+1	< 3.E+1	< 3.E+1	3.39E+2 \pm 47%	< 7.E+1

		--- Gamma Emitters, pCi per gram \pm 1s ---				
		Co-60	Cs-137	U-235	U-238	Am-241
*DHW34008-99	00-30	< 1.E+0	< 1.E+0	2.08E+1 \pm 5%	1.86E+3 \pm 5%	< 1.E+1
*DHW340D08-99	00-31	< 1.E+0	< 1.E+0	1.68E+1 \pm 5%	1.16E+3 \pm 5%	< 2.E+1
**DHW41008-99	00-32	< 3.E+1	< 5.E+1	8.55E+3 \pm 5%	2.23E+5 \pm 10%	< 4.E+2
**DHW410D08-99	00-33	< 3.E+1	< 5.E+1	8.13E+3 \pm 5%	2.13E+5 \pm 5%	< 5.E+2

*green solid
 **yellowcake

Uranium measured by gamma, converted from pCi per gram to μg per gram

U-235 = 2.160 pCi per μg

U-238 = 0.336 pCi per μg

Sample	Lab ID	U-235 $\mu\text{g/g}$	U-238 $\mu\text{g/g}$	*Approximate Enrichment
*DHW34008-99	00-30	9.65E+0 $\pm 5\%$	5.54E+3 $\pm 5\%$	0.17% U-235
*DHW340D08-99	00-31	7.78E+0 $\pm 5\%$	3.46E+3 $\pm 5\%$	0.22% U-235
**DHW41008-99	00-32	3.96E+3 $\pm 5\%$	6.64E+5 $\pm 10\%$	0.59% U-235
**DHW410D08-99	00-33	3.76E+3 $\pm 5\%$	6.35E+5 $\pm 5\%$	0.59% U-235

*green solid

**yellowcake

*These enrichment figures were calculated from the gamma data, and are only approximate.